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Corrosion Behavior of Sacrificial Coatings on Grade 10.9 Fasteners for Multimetal Armor Applications

by Thomas Considine, Thomas Braswell, and John Kelley

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*Oak Ridge Institute for Science and Engineering.

14. ABSTRACT

This experiment examined the effect of accelerated corrosion testing and immersion testing on 13-mm-grade 10.9 bolts. A matrix containing chemical agent resistant coating (CARC) and bare Al 5059, as well as Rolled Homogeneous Armor (RHA) steel plates with CARC that are each bolted together with one bolt of each pretreatment system were subjected to GM9540P in an attempt to evaluate the corrosion prevention properties of each selected coating and pretreatment while simulating the practical applications of bolt-on armor. Five candidate finishes (zinc plating in accordance with the American Society for Testing and Materials [ASTM]-B 633 with hexavalent chromium conversion coating as control) were tested in replicate sets of assemblies. In the immersion phase of testing, each bath was heated to 75 °F, and the salt solution was agitated in order to prevent stagnation and ensure equal heating. Testing was completed over 500 h, with visual and potential inspections at 1, 2, 4, 8, and 24 h, and every subsequent 24 h thereafter. Accelerated corrosion testing was programmed for 120 cycles GM9540 with inspection for corrosion creep at 20 cycle intervals. Corrosion creep in this experiment was defined visually as frosting for the onset of corrosion and red rust in the percentage of the fastener affected yielding separate observations. Each of the pretreatments are assessed and compared in terms of corrosion inhibition.

15. SUBJECT TERMS

alumiplate, bolt-on armor, corrosion, fasteners, hexavalent chromium, immersion, magniplate, trivalent chromium (TCP), bolts nonchromate, hexavalent chrome, grade 10.9 fasteners, bolt-on armor, aluminum electroplate

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1. Introduction

Hexavalent chromium is a widely used and very successful pretreatment, initiating passivation for corrosion inhibition on many various substrates and forms. The hexavalent-based treatments (Cr^{6+}) effectively passivate the surfaces of zinc and zinc alloy electrodeposits with a thin film that provides a number of benefits including color, abrasion resistance, and enhanced corrosion protection. These hexavalent-based pretreatments also possess a unique "self-healing" property. This self-healing refers to the soluble hexavalent chromium compounds contained within the passivation films that will migrate and repassivate any damaged or exposed areas (*1*). However, hexavalent chromium (Cr^{6+}) is known as a human carcinogen and is listed as a hazardous chemical regulated under the Clean Air Act (*2*). It has been designated by the Environmental Protection Agency (EPA) as 1 of the 17 high-priority toxic chemicals.

Although hexavalent chromium has been a key metal used by the military for many applications over the years, it has come under ever increasing scrutiny because of the environmental and human health risks. The Department of Defense (DOD) Under Secretary of Defense for Acquisition, Technology & Logistics, John J. Young Jr., wrote a memorandum on April 8, 2009 addressed to the military service secretaries (*3*). The memorandum calls for more aggressive action to update relevant specifications to authorize the use of qualified substitutes in order to minimize the use of hexavalent chromium. The purpose of this experiment is to identify potential alternative corrosion inhibiting coatings in order to reduce the need and use of hexavalent chromium on fasteners in bolt-on armor applications.

1.1 Experimental Procedure

The selected alternative coating systems were applied to 13-mm-grade 10.9 bolts by the manufacturer prior to testing. Below is a list of the candidate fastener coating systems.

1.2 Test Candidates

The candidate fastener coating systems are listed as follows:

1. Control: American Society for Testing and Materials (ASTM) B633 (*4*) electroplated zinc with hexavalent chromium conversion coating

2. Trivalent Chromium Process (TCP): ASTM B633 (*4*) electroplated zinc with trivalent chromium conversion coating

3. AlumiPlate: Process details, entire surface electroplated with aluminum (Al) alloy 1199 at 99.99 % purity, conversion coated with TCP, then threaded areas coated in accordance with (IAW) MIL-PRF-46010G (*5*) solid film lubricant

4. Magni 565: Process details, entire surface coated with inorganic zinc-rich coating and top coated with Al-rich organic topcoat
5. Magni 594: Process details, entire surface coated with inorganic zinc coating and top coated with a friction modified organic Al-rich coating

For the immersion phase of testing, a 1/8-in hole was drilled into the center of the head of each bolt using a drill press. A Zn-plated steel screw, wrapped with the exposed end of an insulated wire, was screwed into each hole and soldered in place. A polysulfide sealant (Aerospace Material Specification [AMS] 8802B) (6) was applied to each bolt/screw/wire assembly to further insulate and prevent corrosion and contamination of the site of the assembly (figure 1).

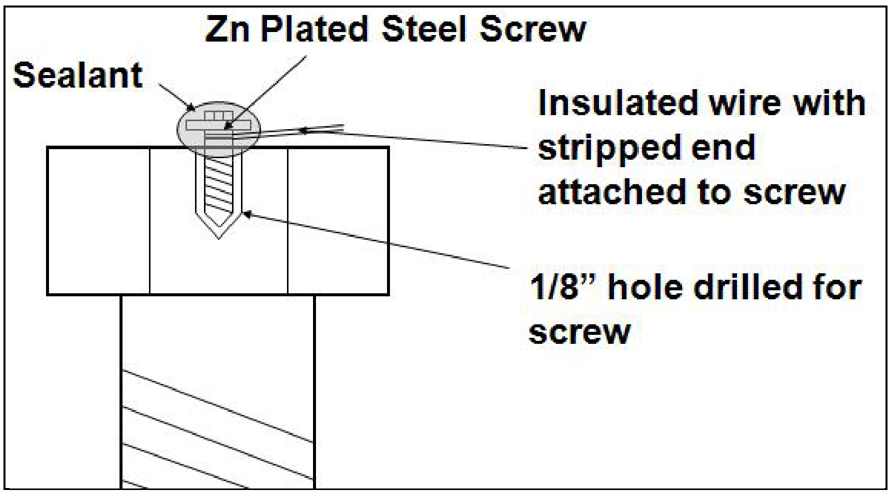

Figure 1. Bolt, screw, wire, and sealant assembly.

The immersion phase of testing involved duplicates of each bolt being placed into a heated salt water bath for a 500-h duration. During the immersion, the open circuit potential of each bolt was continuously monitored vs. Saturated Calomel Electrode (SCE).

A thermostatic bath was filled with a 5% NaCl solution. This bath was heated to 75 °F for the duration of the experiment and was constantly agitated by an internal mechanism. For each bolt candidate and baseline (figure 2), two were immersed in the solution for a period of 500 h. Open circuit potential (OCP) measurements were taken at 1, 2, 4, 8, and 24 h, then every 24 h following that using a digital multimeter.

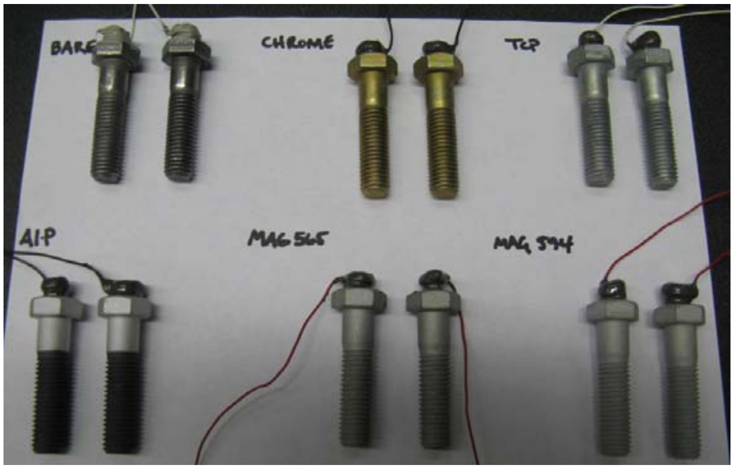

Figure 2. Prepared bolts prior to immersion in thermostatic bath.

Preparation for the GM9540P (*7*) cyclic corrosion tests involved creating test jigs to simulate bolt-on armor. Steel and Al plates were prepared as shown in figures 2 and 3. All of the steel and Al plates had either five 13-mm-diameter *through* holes or five 13-mm *threaded* holes. The face plate of each set was equipped with *through* holes, whereas the base plate was the one equipped with *threaded* holes. Each bolt was placed through the face plate and affixed into the base plate with the *threaded* holes. The plates were approximately 4-in wide by 12-in long and were of various thicknesses. Table 1 shows a list of the plates used in this experiment.

Table 1. Quantity and size of plates used to simulate bolt-on armor.

Quantity	**Plate Thickness (in)**	**Material**	**Hole Type**
6	1/2	RHA Steel	Through
18	1	Al 5059	Threaded
12	1/2	Al 5059	Through

The 12 Al 5059 plates with through holes were abrasive blasted to a 1.5-mil surface finish using 60 grit Al oxide abrasive blasting media. After blasting, each plate was sealed in a corrosion resistant wrap. Additionally, 14 1-in and 10 1/2-in Al 5059 plates were treated with TCP and sealed in a corrosion resistant wrap. All six RHA steel plates (*6*) of the 1-in Al 5059 threaded plates with TCP and five of the 1/2-in Al 5059 plates were primed with MIL-DTL-53022-10 (*8*) to a dry film thickness (DFT) of 2.0–2.5 mils. After being allowed to dry for 24 h at ambient room temperature, these panels were top coated with MIL-DTL-53039 B (*9*) type II chemical agent resistant coating (CARC) with a DFT of 1.8–2.2 mils. This set of panels was then cured at room temperature and 50% relative humidity for 168 h.

Figure 3. RHA steel prior to surface finishing.

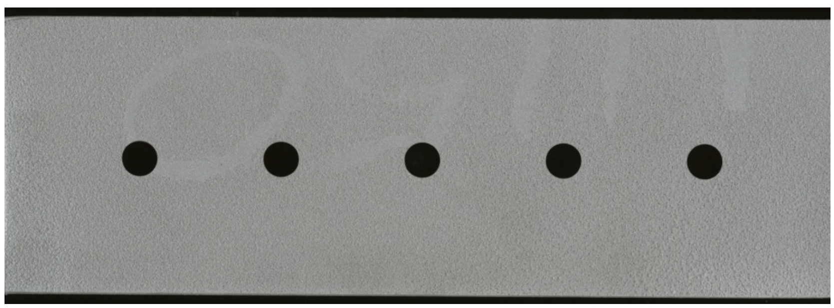

Figure 4. RHA steel specimen after abrasive blasting.

The 1/2-in plates were then bolted to the 1-in plates to create an array of six specific groups. Each assembly used one bolt from each coating system to be evaluated: hexavalent chrome, TCP, AlumiPlate, Magniplate 565, and Magniplate 594. The first group contained five sets of Al 5059 1 in threaded plates bolted to Al 5059 1/2-in plates with through holes. Each set in group 1 had received abrasive blast, TCP treatment, and a CARC system. The second group contained five sets of Al 5059 1 in threaded plates bolted to Al 5059 1/2-in plates with through holes. Each set in group 2 had received an abrasive blast and TCP treatment but were unpainted. The third group contained two sets of Al 5059 1 in threaded plates bolted to Al 5059 1/2-in plates with through holes. Each set in group 3 had only received the abrasive blast. The fourth group contained two sets of Al 5059 1-in threaded plates bolted to RHA steel 1/2-in plates with through holes. Each set in group 4 had received an abrasive blast, TCP treatment, and a CARC system. The fifth group contained two sets of Al 5059 1 in threaded plates bolted to RHA steel 1/2-in plates with through holes. Each set in group 5 had received the abrasive blast and TCP treatment but were unpainted. The sixth group contained two sets of Al 5059 1 in threaded plates bolted to RHA steel 1/2-in plates with through holes. Each set in group 6 had only received the abrasive blast. The full description of the sample arrays can be seen in table 2. For example, group 4

consists of two replicates of this combination: one AL 5059 (1-in-thick) plate that was blasted, pretreated with TCP, and CARC coated, coupled with one RHA Steel (1/2-in-thick) plate that was blasted, pretreated with TCP, and CARC coated. All groups have one of each bolt (figure 5)

Table 2. Description of the bolt-on armor combinations.

		Threaded 1-in Al 5059			**Unthreaded 1/2-in Al 5059**			**Unthreaded 1/2-in RHA Steel**		
Group ID	**Qty**	**Blast**	**TCP**	**CARC**	**Blast**	**TCP**	**CARC**	**Blast**	**TCP**	**CARC**
I	5	√	√	√	√	√	√			
II	5	√	√		√	√				
III	2	√			√					
IV	2	√	√	√				√	√	√
V	2	√	√					√	√	√
VI	2	√						√	√	√

Figure 5. Photograph of plate assembly prior to final assembly (from left to right: Magniplate 594, Magniplate 565, Alumiplate, TCP, Hexavalent chrome).

Each bolt was hand fastened into the test plates, using a box wrench when necessary, using its corresponding washer. A calibrated torque wrench was then used to apply a 60-in-lb load on each of the seated fasteners. The plate assemblies were labeled 1–18, and each of the fasteners was labeled on the surface of the plate for easy identification. Each plate assembly was placed into a GM 9540P test chamber at an angle that was dictated by the protrusion of the fastener from the back of the assembly. Care was taken to ensure that the angle for all assemblies was similar (figures 5 and 6).

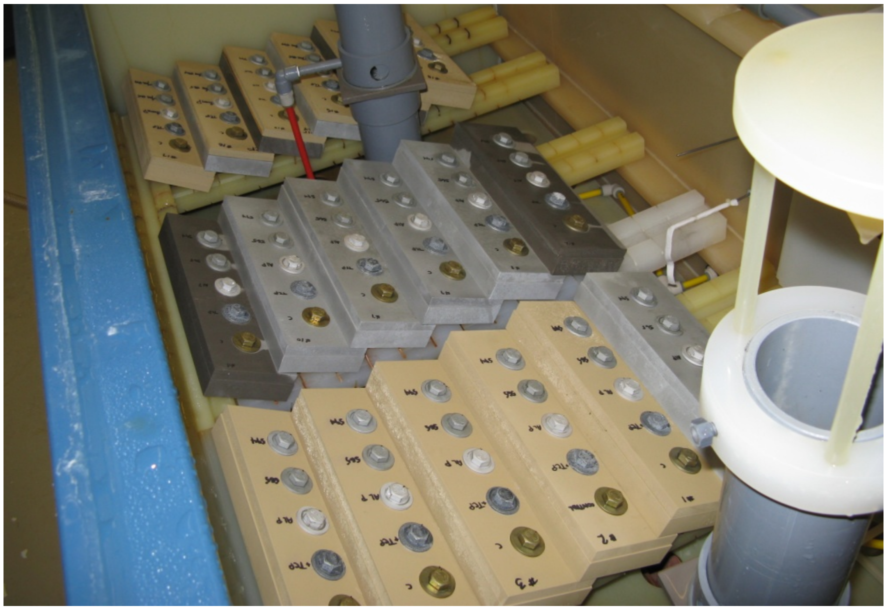

Figure 6. Bolt-in-plate arrays loaded in GM9540P accelerated corrosion chamber prior to testing.

Observations and evaluations were made at 20-cycle intervals to identify the percentage of the fastener head and washer that had either begun "frosting" prior to the onset of the steel/iron in the fastener, as well as the percentage of the fastener and washer that had been affected by "red rust" (examples of which can be seen in figure 7). After the full duration of 120 cycles, each fastener was removed from the assembly using a calibrated torque wrench, and the break-away torque was recorded.

Figure 7. Examples of "frosting" and "red rust."

2. Results and Discussion

2.1 Immersion Phase

The percentage of corrosion observed on each bolt following 500 h of immersion is plotted in figure 8. Visually, the corrosion rate of the TCP-coated bolt was much higher than that of all other coatings tested, scans of which are presented in figure 9.With respect to the hexavalent chromium baseline, only the TCP and uncoated steel had a higher percentage of visual corrosion over the same time period. 100% of the surface area of the TCP treated bolts had corroded approximately within 150 h in contrast to the untreated bare steel bolt, which lasted approximately 250 h before reaching total surface corrosion. The AlumiPlate-coated bolt never exceeded 40% corrosion of the total surface area, whereas the Magniplates never exceeded 20%. These fasteners were among the most corrosion resistant in the total immersion experiment, having the lowest total corrosion after 500-h immersion. By contrast, 80% of the surface area of the hexavalent chromium had corroded by the end of the experiment.

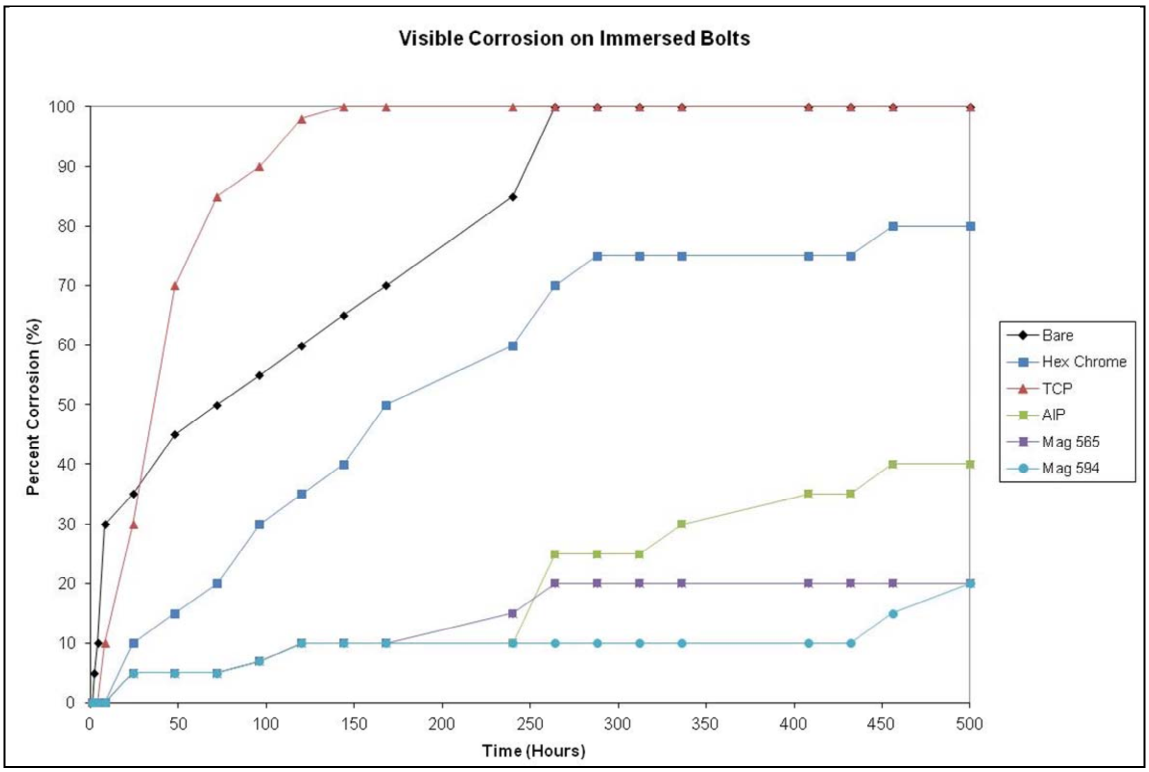

Figure 8. The amount of corrosion observed visually on the immersed bolts samples during periodic measurements over 500 h.

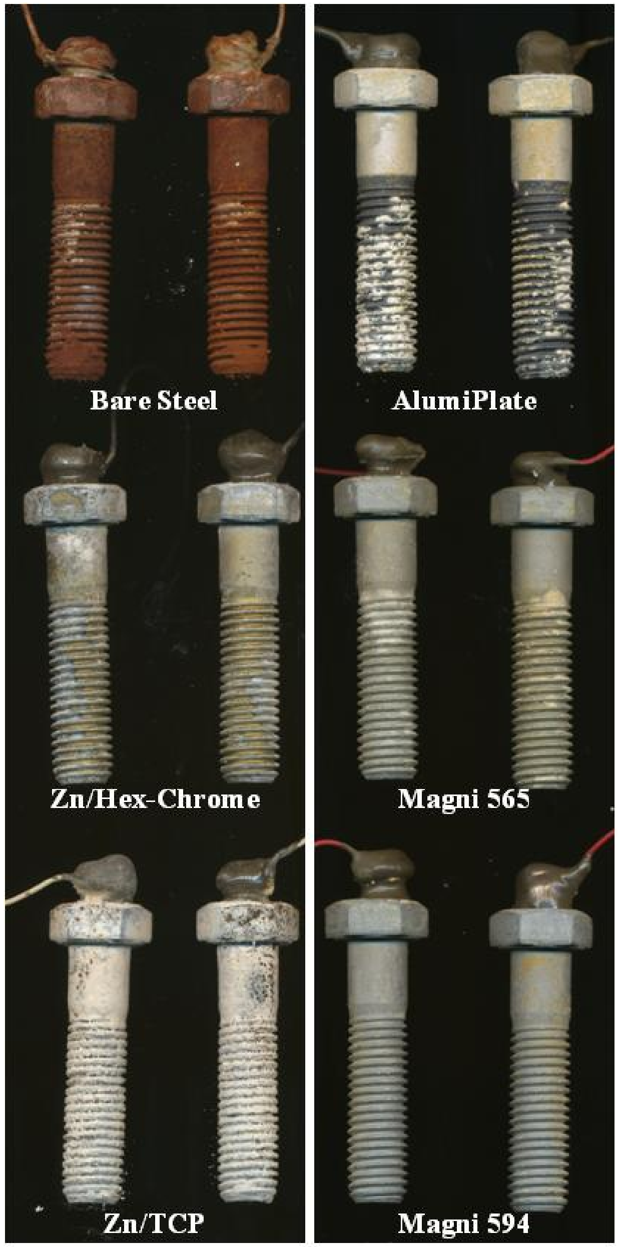

Figure 9. Bolts after 500-h immersion (from top to bottom, left to right: bare steel, Alumiplate, Cr^{6+}, Magni 565, TCP, Magni 594).

The open circuit potential of the bolts was monitored while immersed in the heated 5% NaCl solution over a period of 500 h. Figure 10 illustrates the changes in OCP throughout the test. Both the uncoated steel and the hexavalent chrome experienced very little change over the course of the 500-h immersion. The Magniplate 594 underwent a very large drop of about 0.25 V in the first 24 h. However, the OCP of the AlumiPlate bolts significantly increased by more than 0.20 V over the first 24 h before dropping back down after 100 h to its original 0.8 V before dropping down to 0.76 V at the end of 500 h. At the nominal voltage (nV) of OCP, the

Magniplate 565 also began to drop, but not as sharply as the AlumiPlate (approximately 0.50 V), but experience a steady drop in OCP. Magniplate 565's total drop after 500 h was 0.15 V. The TCP and hexavalent chrome-coated bolts followed an almost identical trend until they reach about 250 h of immersion, after which the OCP of the TCP began to drop steadily. Visually, the most prominent difference between TCP and Cr^{6+} was the amount of surface corrosion. At the 250-h point, the TCP coating was 100% corroded, whereas the hexavalent chromium was approximately 60% corroded. From the 250-h mark until the end of testing at 500 h, the TCP dropped approximately 0.20 V.

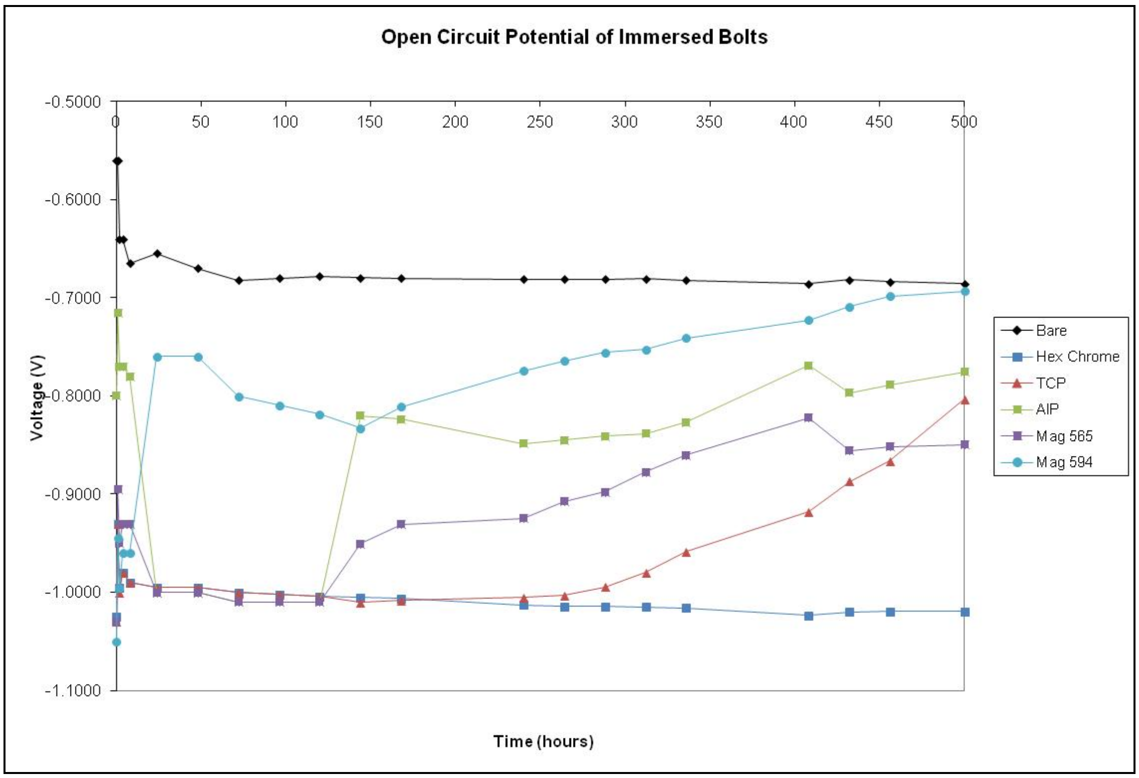

Figure 10. The open circuit potential of the immersed bolt samples over 500 h.

2.2 Accelerated Corrosion

The most notable outcome of the accelerated corrosion testing of the bolt-in-plate assemblies was that the variations in mated plates and coating systems appeared to have no influence on to the relative corrosion rate of the bolts. In other words, the bolts corroded at the same rate, relative to each other in GM9540P as they did in the immersion experiment. As with the immersion experiment, it became evident after only 20 cycles of GM9540P that the TCP fasteners failing prematurely. These bolts began to leach zinc after only a few cycles, and by 20 cycles had already begun to show signs of steel corrosion or red rust (figure 11). Some of this corrosion occurred at sites damaged by the wrench during assembly, specifically the edges of the bolt heads. However, much of the visible red corrosion occurred on surfaces that were initially

pristine. After 60 cycles of GM9540P, the TCP-coated fastener was between 60% and 100% corroded, after which point no further corrosion was observed. Passivation films on electroplated zinc deposits have been successfully used commercially for many years. Their reliability and performance has improved with time, allowing many end users to change their zinc plating specification requirements in favor of this more environmentally aware technology (*1*) The excessively poor performance of the TCP sealed bolts used in this experiment is uncharacteristic of what is typically seen with TCP. It is likely that the TCP sealing process was inadequately conducted, which led to the application of an inferior coating on the bolts.

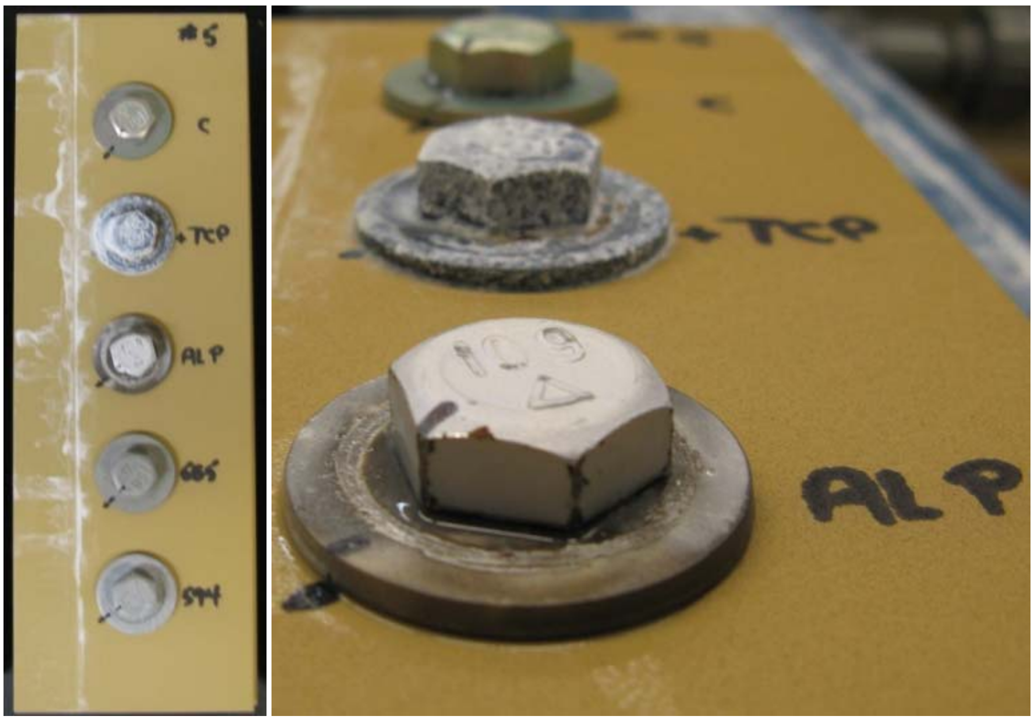

Figure 11. Representative of the bolt-in-plate sample showing an array after GM9540P exposure of 20 cycles, closeup showing the condition of the TCP and AlumiPlate bolts after 20 cycles.

The AlumiPlate fasteners also showed signs of red corrosion products by 20 cycles. Unlike the TCP sealed bolts, these sites were confined to the edges of the heads of the bolts and other areas of the coating that were damaged during assembly. Over the next 100 cycles, the AlumiPlate fasteners exhibited very little additional corrosion, and the corrosion did not extend much beyond the original corrosion sites. The Magniplate and AlumiPlate fasteners, in general, performed very well as compared to the baseline hexavalent chromium fasteners.

Only the TCP bolts exhibited corrosion in excess of 10% of the total surface area evidenced by the graphs of the percent visible corrosion of the bolt heads and washers in the bolt-in plate in appendix B. All of the other coatings provided adequate protection to the steel bolts.

After the completion of the accelerated corrosion testing, the bolt-in-plate assemblies were disassembled in order to examine any mechanical effects that may have occurred due to corrosion. The break-away torque was measured during the disassembly in addition to observing the relative ease of removal. A bolt was labeled as *easy* if it could be removed by hand after the initial break away. A bolt was labeled as *moderate* if its removal required the use of a wrench after the initial break away but did not offer much resistance. Finally, bolts that required the use of a wrench after the initial break away but offered heavy resistance to removal were labeled as *hard.* The values for break-away torque and the color designation for ease of removal can be seen in table 2. In terms of ease of removal and break-away torques, the Magniplate coatings appear to have the most lubricious properties. With the initial torque of 60-in lbs, the Magniplate 565 had the lowest average break-away torque of 29-in lbs, and it was the easiest to remove by hand. Magniplate 594 was very similar with only two moderately difficult fasteners out of the set of 18 and with a slightly higher average break-away torque than the Magniplate 565 at approximately 32.5-in lbs. It is important to note that the baseline hexavalent chrome had an average break-away torque value of approximately 51-in lbs. In most applications, it is not desirable to have the break-away torque drop substantially while in service. This can indicate that the fasteners in loosening over time and in service, could cause a failure. Only the AlumiPlate bolts had an average break-away torque similar to the baseline and a standard deviation that was even lower. Although the AlumiPlate fasteners were more difficult to remove than the MagniPlate fasteners, they are more comparable with what is expected in the baseline fasteners. Because of the excessive corrosion that occurred in the treads of the TCP fasteners, they were the most difficult to remove having the highest average break-away torque at approximately 76-in lbs. As stated earlier, the bolts were loaded prior to the experiment at 60 psi. As can be seen in table 3, the loads tended to decrease over the course of the experiment, with the exception of the TCP-coated fasteners.

Table 3. Break-away torques and ease of removal.

Plate	HEX	TCP	ALP	565	594
1	35	55	37	20	32
2	36	47	47	29	34
3	44	52	40	28	33
4	54	65	39	30	29
5	35	56	61	29	28
6	56	80	65	35	43
7	66	85	65	38	41
8	60	76	62	37	35
9	54	79	80	36	41
10	57	131	61	39	40
11	50	110	57	36	41
12	57	111	63	39	39
13	68	90	60	24	33
14	53	100	74	35	37
15	50	63	57	9	31
16	56	66	48	32	32
17	45	62	46	25	14
18	40	36	0	2	3
avg	50.9	75.8	53.4	29.1	32.6
std dev	9.9521732	25.119843	17.796967	10.223803	10.007187

EASY
MODERATE
HARD

3. Conclusions

The Magniplate samples provided the best protection against corrosion for the steel fasteners. For both the total immersion and the GM9540P tests, MagniPlate had the least amount of visible corrosion. The MagniPlate and AlumiPlate bolts outperformed the baseline zinc plated with hexavalent chromium sealed bolts.

In fact, with the exception of the TCP sealed zinc plate, all of the potential alternatives tested provided as good or better corrosion protection to the bolts as the hexavalent chromium sealed zinc plate. Only the TCP sealed zinc-plated bolts had more than 10% of the surface area of the bolts affected by corrosion.

The Magniplate-coated fasteners had the lowest average break-away torque indicating that the coating has some residual lubricity. Although these bolts were the easiest of all the coated fasteners to remove, it may not be the most desirable property for mission critical components, because it would allow fasteners to vibrate loose. The break-away torque for the AlumiPlate bolts was very similar to that of the baseline chromate sealed zinc-plated bolts. Because Alumiplate provided better than baseline corrosion protection, and will likely sustain torque values in the field, it is considered to be the best choice of the coatings tested to replace the chromate zinc-plated bolts. Magniplate-coated fasteners can be considered but only when used

with a thread locking adhesive. Additional tests would need to be conducted to determine which adhesive would be most appropriate.

The excessively poor performance of the TCP sealed bolts was uncharacteristic of what is broadly considered to be an affective post treatment. It is the author’s opinion that the process used for applying the TCP sealer was inadequately conducted and is what led to the premature failure of these bolts. It is recommended that further studies be done using TCP sealers that have been properly applied under controlled conditions. Also, additional validation should be performed on the AlumiPlate to determine their potential compatibility with multimetal and mixed metal assemblies in military hardware before recommending implementation.

4. References

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Appendix A. Additional Photographs of Test Samples

Figure A-1. Plates 1–3 disassembled after 120 cycles GM9540P.

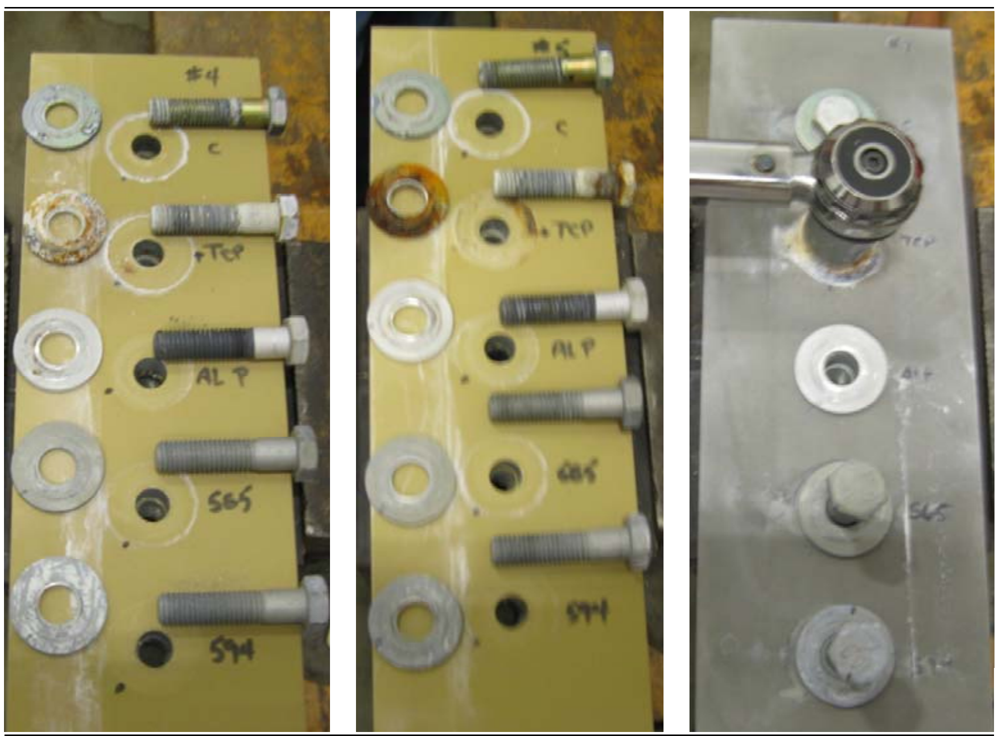

Figure A-2. Plates 4–6 disassembled after 120 cycles GM9540P.

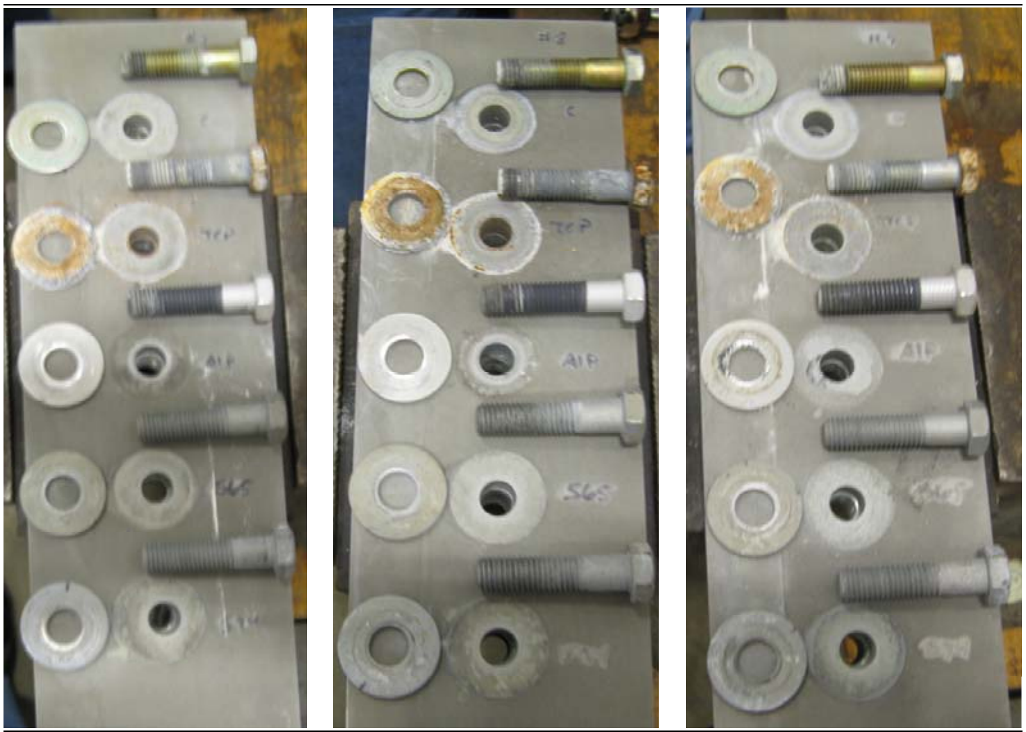

Figure A-3. Plates 7–9 disassembled after 120 cycles GM9540P.

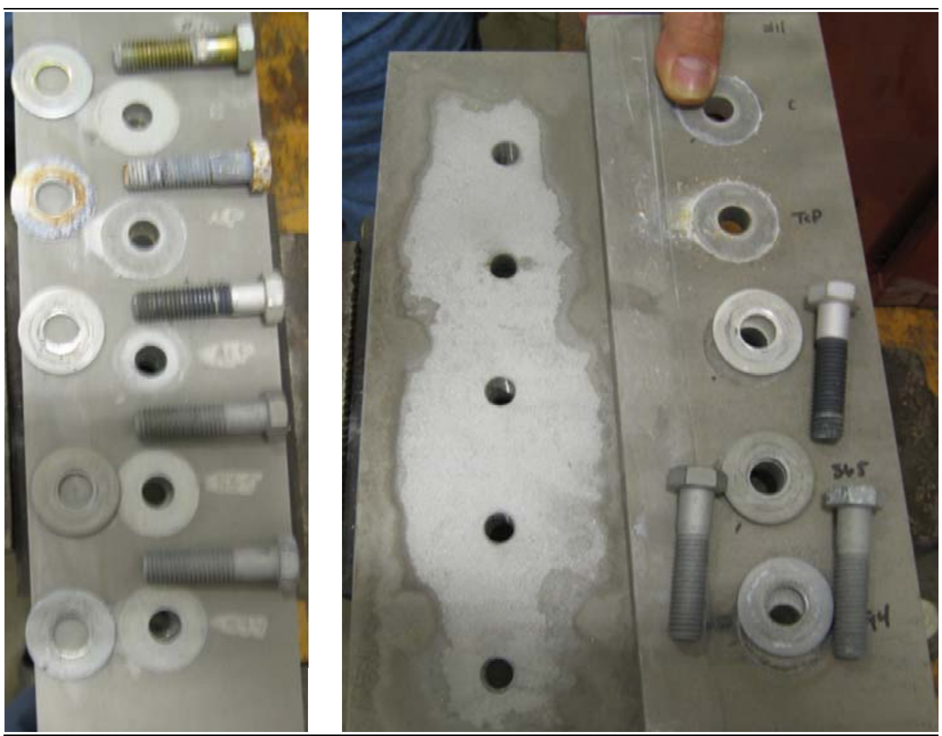

Figure A-4. Panels 10 and 11 disassembled after 120 cycles GM9540P (including interior shot to show moisture penetration on panel 11).

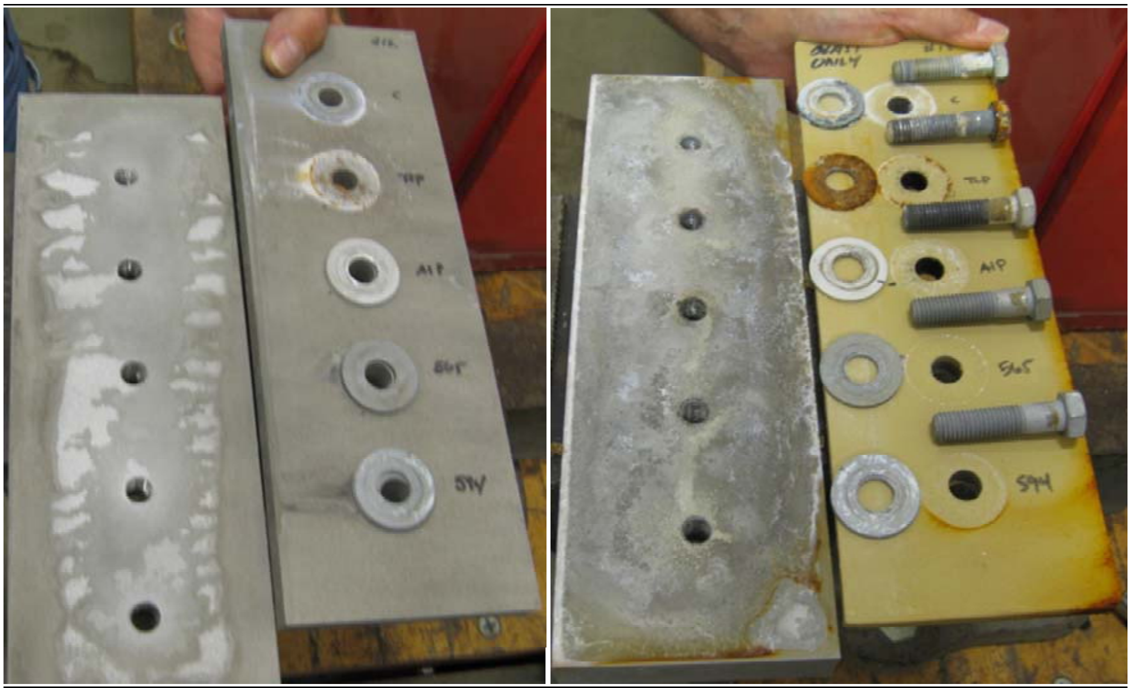

Figure A-5. Panels 12 and 13 disassembled after 120 cycles GM9540P (including interiors to show moisture penetration).

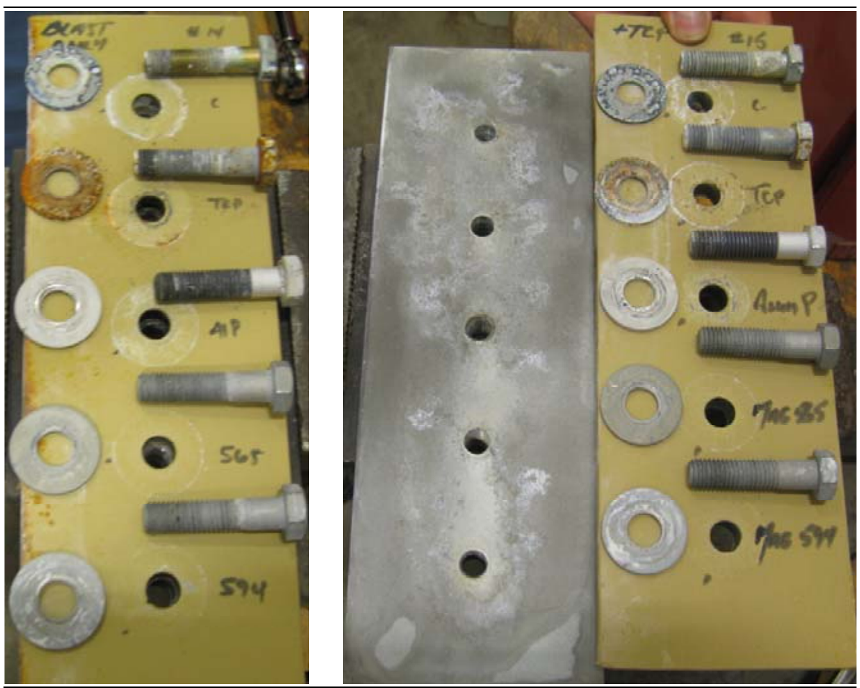

Figure A-6. Panels 14 and 15 disassembled after 120 cycles GM9540P (including interior shot to show moisture penetration on panel 15).

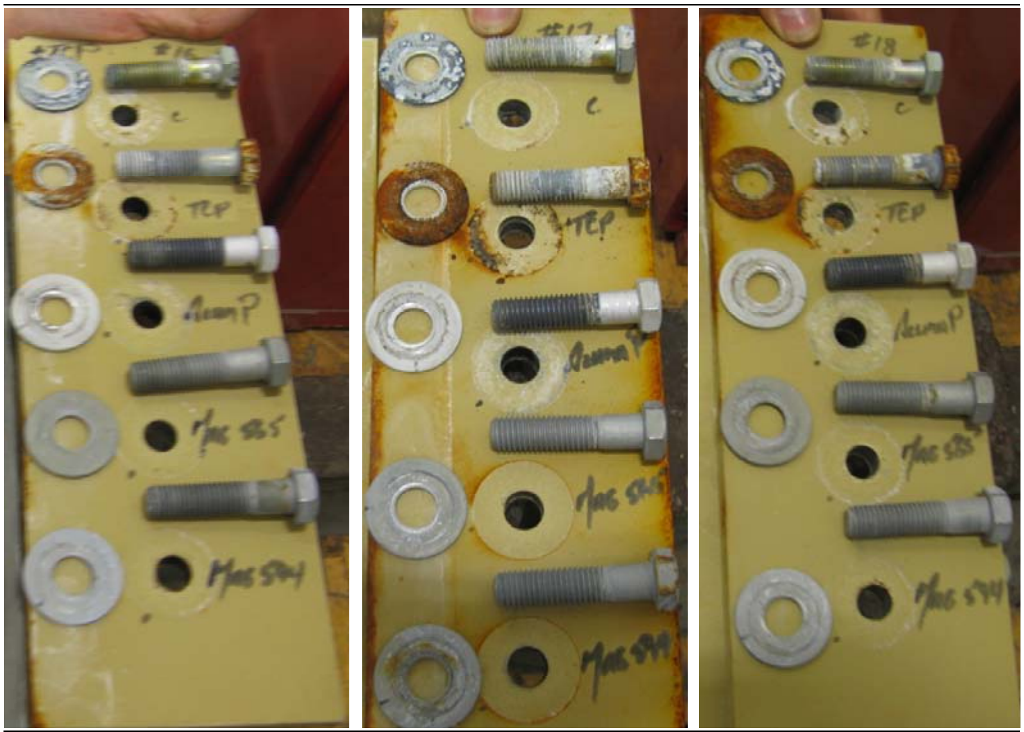

Figure A-7. Panels 16, 17, and 18 disassembled after 120 cycles GM9540P.

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Appendix B. Results for the Bolt-in-Plate in 9540P Cyclic Corrosion Tests

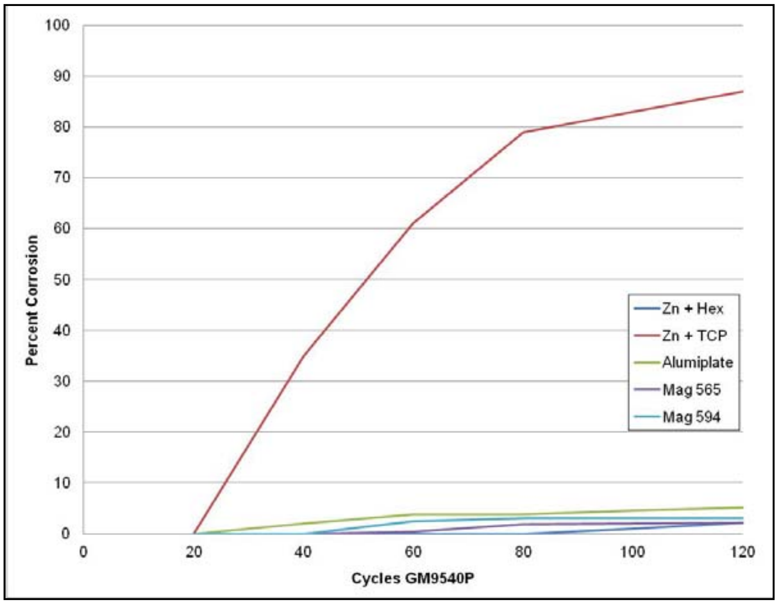

Figure B-1. Group I combination; Al5059 (abrasive blast-TCP-CARC) / Al5059 (abrasive blast-TCP-CARC).

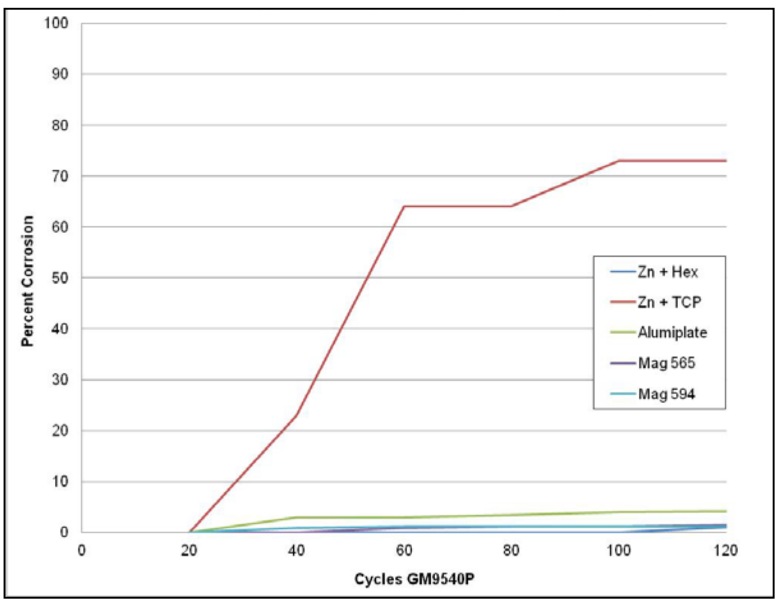

Figure B-2. Group II combination; Al5059 (abrasive blast-TCP) / Al5059 (abrasive blast-TCP).

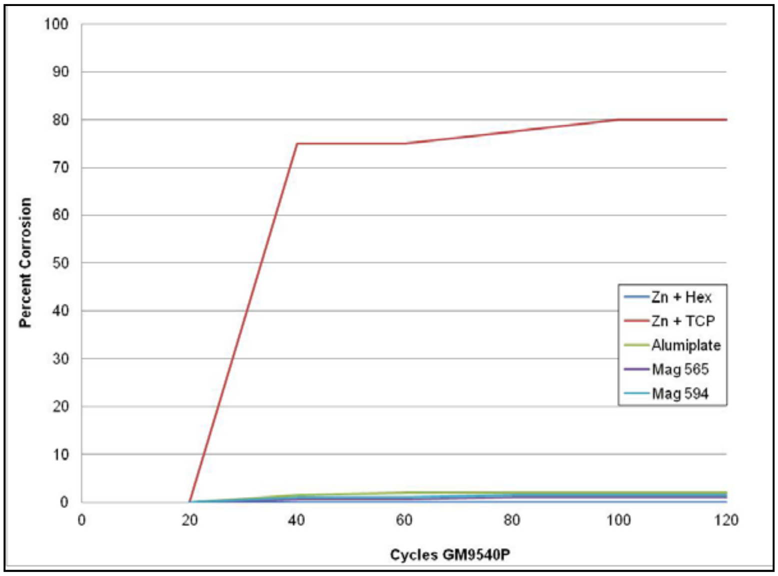

Figure B-3. Group III combination; Al5059 (abrasive blast only) / Al5059 (abrasive blast only).

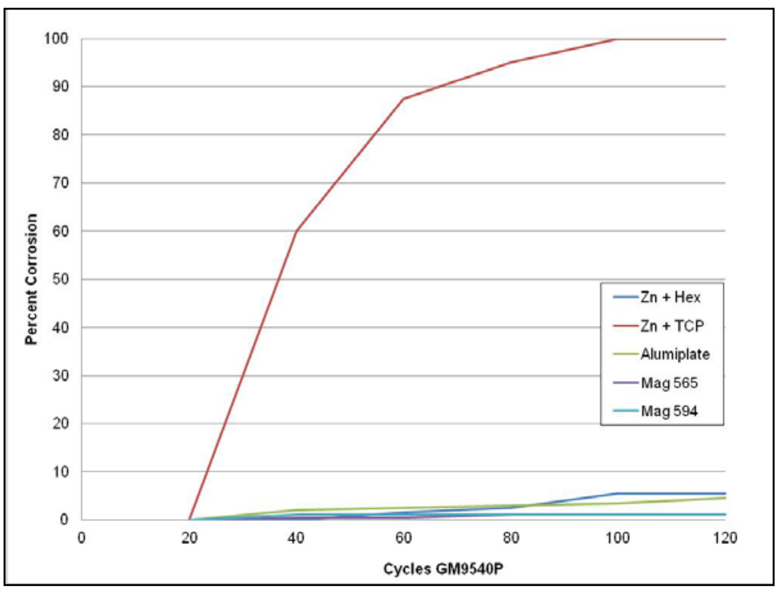

Figure B-4. Group IV combination; Al5059 (abrasive blast-TCP-CARC) / RHA steel (abrasive blast-TCP-CARC).

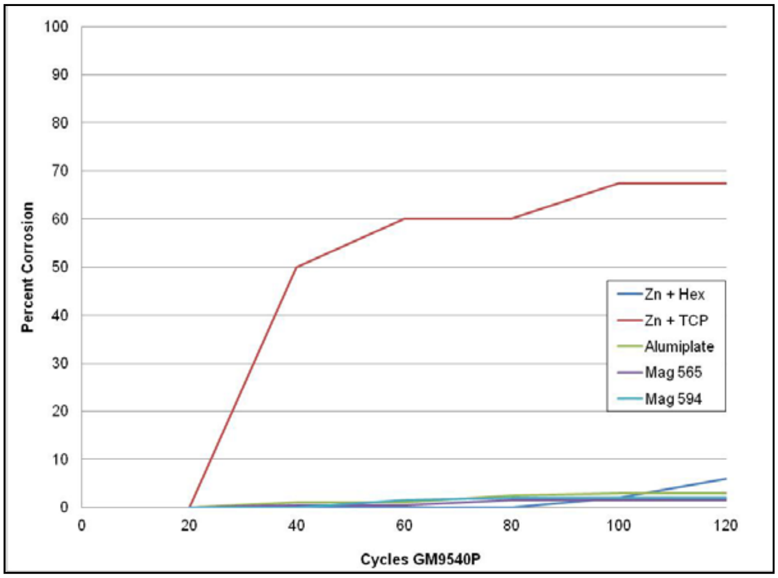

Figure B-5. Group V combination; Al5059 (abrasive blast-TCP) / RHA steel (abrasive blast-TCP-CARC).

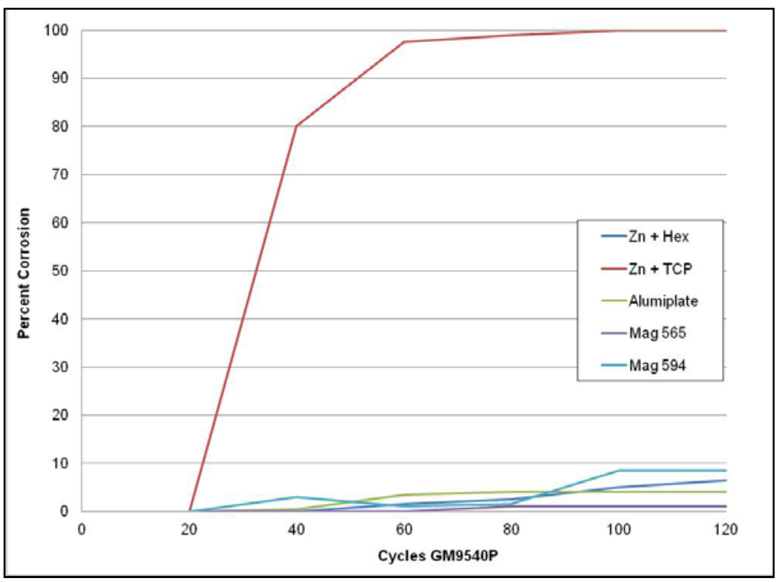

Figure B-6. Group VI combination; Al5059 (abrasive blast only) / RHA steel (abrasive blast-TCP-CARC).

List of Symbols, Abbreviation, and Acronyms

ALP	AlumiPlate, Electroplated Aluminum
Al 5059	Aluminum Alloy 5059
ASTM	American Society for Testing and Materials
Blast	Abrasive Blasted
DOD	Department of Defense
CARC	Chemical Agent Resistant Coating
EPA	Environmental Protection Agency
HEX	Hexavalent Chrome
nV	nominal voltage
OCP	Open Circuit Potential
RHA	Rolled Homogeneous Armor
SCE	Saturated Calomel Electrode
TCP	Trivalent Chrome Process

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